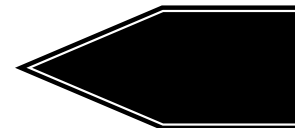


Lab Demo - 7&8



7. High Volume Sampler for Measurement of SPM, RSPM, SO_x & NO_x

All micro-organisms responsible for causing the large number of pathogen-caused respiratory, eye and skin diseases, various allergens, fibrous materials, heavy metals, metallic fumes and even many organic carcinogens are present in air in the form of suspended particulates. It is for this reason that TSPM (Total Suspended Particulate Matter), SPM (Suspended Particulate Matter) or RSPM (Respirable SPM) in air has been world-over considered a "Criteria Parameter" to indicate air quality.

Practically all fuels in common use contain variable amount of sulphur, most of which is discharged to the atmosphere as SO₂ during combustion. In addition, specific industrial processes produce large quantities of SO₂, some of which may escape into the air. The concentration to be found in the atmosphere is governed not only by the number and size of local sources but by factors, such as the height of chimneys and meteorological conditions. Pollution of the air by SO₂ is widespread since it exists wherever fossil fuels are burned. It is harmful to stonework & metal work; it can damage plant tissues, and may aggravate such illnesses as bronchitis. High Volume Air Samplers based on the simple design standardized by USEPA have been in use all over the world for monitoring the TSPM, SPM and RSPM in ambient air.

Principle:

The High-Volume (Hi-Vol) Sampler or Respirable Dust Sampler is the workhorse of air sampling and monitoring. The sampler uses a continuous duty blower to suck in an air stream. When fitted with a particle size classifier, it separates particles greater than 10µm size from the air stream. The air stream is then passed through a filter paper to collect particles lesser than 10µm size (PM₁₀). Gravimetric measurements yield values of suspended particulate matter (SPM), as the sum of the two fractions, and PM₁₀, the material retained on the filter paper. The filter paper can be used to determine benzene-soluble organics, metals, such as Pb, Cd, etc., fluorides, radioactive materials and biologically active non-metals, sulphate, nitrate and ammonium.

The sampler can also be used to sample gaseous pollutants. A stream of unfiltered air is bubbled through a reagent, which either reacts chemically with the gas of interest or into which the gas is dissolved. Wet chemical techniques are then used to measure the concentration of the gas. The Hi-vol sampler uses a cyclone type particle size classifier, illustrated in Figure 7.1, to separate out particles of diameter greater than 10µm aerodynamic diameter. Ambient air laden with SPM enters the cyclone near its top where the air stream is given a swirling motion. The resulting centrifugal acceleration moves the coarse and heavier air borne particles to the outer wall. These separated particles fall

through the cyclone conical hopper and are collected in the sampling bottle placed at the bottom. The air containing the respirable dust exits through a cylindrical outlet, mounted concentrically at the top of the cyclone, and enters the filter assembly.

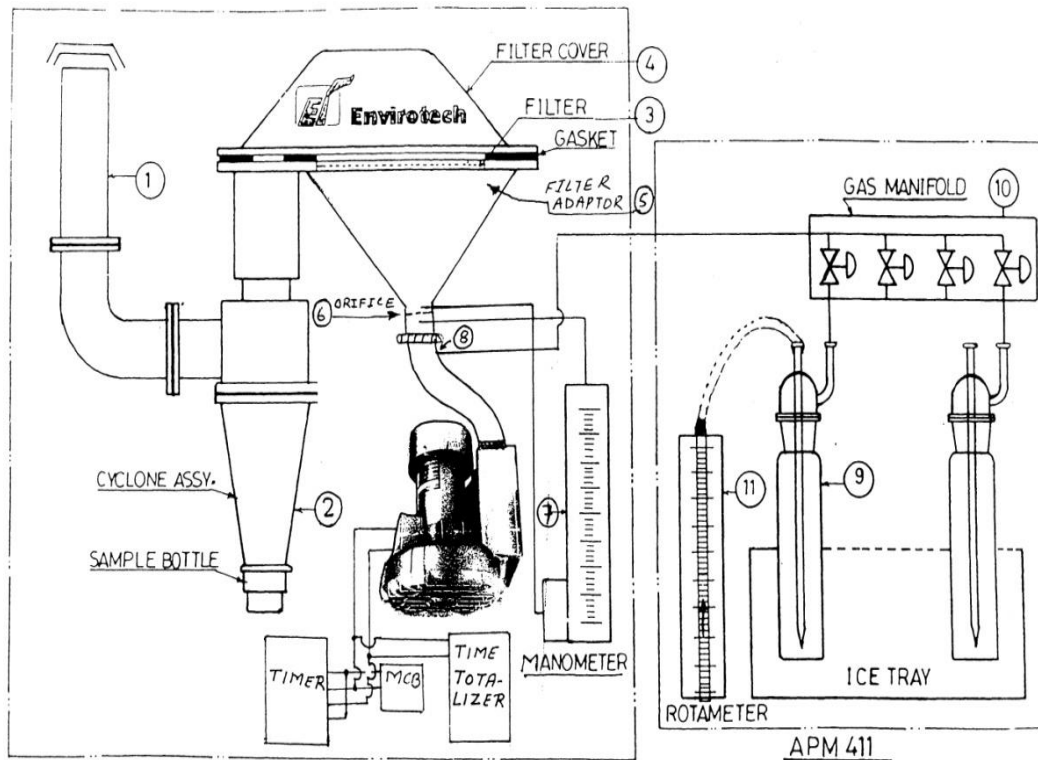


Figure 7.1: The schematic of the High Volume Sampler

Operation of Sampler:

1. Check that pre weighted fresh filter is mounted in the filter holder assembly.
2. Check that manometer fluid is filled up to zero mark
3. Feed the duration of sampling in the timer after setting real time and day.
4. Record initial time totalizer reading.
5. Shift left timer button on auto position and shift right button on run position.
6. Blower shall start and manometer fluid will rise. Let water level stabilize and record initial manometer reading in m³/min after 5 minute of operation.
7. In most cases only a gravimetric analysis of the Respirable dust and Total Suspended Particulates will be attempted. This involves weighing of the filter to determine the quantity of respirable dust deposited on it and a weighment of the dust retained by the cyclone to assess the TSPM concentration.

8. To avoid the effect of moisture absorption on the filter it must be suitably conditioned in a desiccator at room temperature for 12 to 16 hours prior to weighing.
9. Gaseous pollutants are absorbed in suitable absorbing solutions placed in the impinger bottles. The absorbing reagents are recovered from the impingers and analyzed in the laboratory.

Precautions:

1. Glassware used in analysis need to be cleaned properly
2. Chemicals whose strength change with time need to be prepared fresh (e.g. sodium metabisulphide)
3. Reagents used for analysis must be brought to same temperatures before mixing.
4. Chemicals need to be stored as per recommendation (Ambered bottles, away from heat and light)
5. Dry chemicals should be transferred from bottles using a clean spatula.
6. Reagent bottles should be properly tightened.
7. Prepared chemicals should not be left in the volumetric glasswares. These should be transferred to reagent bottles.
8. Always use analytical grade reagent for air pollution monitoring
9. Check quality of distilled water regularly.

8. Stack Sampling and Gaseous Emission Monitoring

Stack Sampling

Monitoring of Stack and vent emissions is now becoming a routine requirement not only for large but even the medium and small industrial units. There has also been a growing realization that gaseous pollutants, chemical fumes and fine rusts are hazardous as particulate ashes and dusts. Stack sampling is a direct measurement of PM and Gaseous pollutant concentration at the point of release. Emission sampling is performed at specified time intervals with specific reporting requirements to verify compliance.

Principle:

Flue gases enter the system through the nozzle at the tip of the sampling probe, pass through the filter thimble, where particulate matter (PM) is removed and reach the sampling train/condenser assembly in the cold box section of the instrument panel. Here the gas stream is split into two sections. One section passes at low flow rate (0.5- 3 LPM) through a train of impingers loaded with suitable reagents to absorb gaseous pollutants, relevant to the emission source while the remaining gas stream bubbles through a distilled water impinger followed by silica gel. On passing through the cold box section, the flue gases cool down, releasing any moisture or condensable present and are scrubbed for corrosive or toxicant fractions. Relatively clean gases then pass through the flow meter and dry gas meter so that the volume of flue gas sampled is measured and are subsequently exhausted into the atmosphere through the vacuum pump.

Provisions are made to accurately measure the pressure drop across the thimble and sampling train assembly using a vacuum gauge. Similarly the temperature of the gas stream near the flow meter inlet can be measured by a pyrometer. Hence the flue gas sample volume can be normalized as per gas laws. Change in weight of the filter is used to determine the quantity of dust contained in the flue - gas sample while a product of the sampling rate and time is used to measure the sample volume.

The basic properties of various gaseous pollutants are used to absorb them in suitable chemical reagents. A filtered sample of flue gas is bubbled through an impinger train at a metered flow rate. The impingers are filled with appropriate reagents that would absorb the gases of interest from the process being monitored. The system allows two gases to be sampled simultaneously. While the volume of gas sampled is determined from the knowledge of the sampling time and flow rate, concentration of individual pollutants must be determined through an analysis of the absorbers.

Since particles in motion have inertia, if the PM concentration in the sample drawn from the stack is to truly represent the PM concentration in the stack, isokinetic conditions must be maintained at the tip of the sampling probe. Apparently non-isokinetic conditions tend to cause a separation of particles and gas molecules so that both the concentration and size distribution are altered by non-isokinetic sampling. Heavier/larger particles are more likely to be affected with lighter/finer particles behaving almost like gas molecules.

Selection of Filter Medium:

Temperature of the flue gases is the major factor in determining/selection of the type of the filter medium to be used. The sampling can be done in two ways. In the first method the filter holder is kept inside the stack. As a result the thimble will be at the same temperature as that of the flue gases. This is the preferred technique since it minimizes deposition of particulates along probe walls and also ensures that any condensable that may be present do not tend to clog the filter medium. The filter holder must be kept inside the stack when sampling for aerosols like acid mist etc., is being attempted. Cellulose filtration thimbles can be used for "IN - Stack" sampling of flue gases upto a stack temperature of 120°C while glass microfiber thimbles allow operation upto 500°C.

In the second method the filter holder is kept outside the stack. A stainless steel union is used to mount a nozzle at the front end of the probe pipe. The filter holder is mounted on the rear end. The flexible hose is connected to the filter holder through a small probe pipe (about 30cm long).

Two precautions need to be kept in mind while using this method.

- After each sampling the probe-pipe must be back washed with a mild solvent like acetone to obtain the particulates that may have been deposited on the probe walls. These particles must then be recovered by

evaporating the solvent and their weight added to that of particles collected on the thimble.

- While sampling flue gases containing moisture or condensable, care must be taken to minimize the length of stainless steel probe outside the stack so that flue gases do not cool down below their dew point resulting in significant amount of condensation before the filter assembly.

Salient Features

Parameter	Specification
Stack Temperature Range	Ambient to 600°C
Stack Velocity Range	0 to 60 m/sec
Manometer Range	0-1300 mm of H ₂ O
Particulate Sampling	2-30 1pm collection on thimble type filters up to 0.3 micron rating
Gaseous Sampling	0.2-3 1pm collection in a set of Borosilicate glass impingers
Rotameter	0-30 1pm for particulate and 0-3 1pm for gases

Equipment for Stack Monitoring:

Monitoring of Stack and vent emissions is now becoming a routine requirement not only for large but even the medium and small industrial units. There has also been a growing realization that gaseous pollutants, chemical fumes and fine rusts are hazardous as particulate ashes and dusts. Stack sampling is a direct measurement of PM and Gaseous pollutant concentration at the point of release. Emission sampling is performed at specified time intervals with specific reporting requirements to verify compliance.

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Two precautions need to be kept in mind while using this method:

- After each sampling the probe-pipe must be back washed with a mild solvent like acetone to obtain the particulates that may have been deposited on the probe walls. These particles must then be recovered by evaporating the solvent and their weight added to that of particles collected on the thimble.
- While sampling flue gases containing moisture or condensable, care must be taken to minimize the length of stainless steel probe outside the stack so that flue gases do not cool down below their dew point resulting in significant amount of condensation before the filter assembly.

Salient Features:

Parameter	Specification
Stack Temperature Range	Ambien 600°C
Stack Velocity Range	0 to 60 m/sec
Manometer Range	0-1300 mm of H ₂ O
Particulate Sampling	2-30 1pm collection on thimble type filters up to rating
Gaseous Sampling	0.2-3 1pm collection in a set of Borosilicate glass impingers
Rotameter	0-30 1pm for particulate and 0-3 1pm for gases

Gaseous Emission Monitoring**i) UV Photometric Ozone Analyzer:**

Using UV Photometric technology, the ozone analyzer measures the amount of ozone in the air from ppb levels up to 200ppm. The ozone analyzer is a dual cell photometer; the concept adopted by the NIST for the national ozone standard. The model has the following features:

- High sensitivity
- Fast response time
- Internal Ozone calibrator
- Linearity through all ranges
- Automatic temperature and pressure compensation

Principle:

Ozone exhibits a strong absorption band in the ultraviolet region at 254 nm. This feature is the basis of the photometric measurement method for ozone. The degree to which the UV light is absorbed is directly related to the ozone concentration as described by the Beer Lambert Law:

Where:

$$I/I_0 = e^{-KLC}$$

K= molecular absorption coefficient, 308 cm⁻¹(at 0°C and at 1 atmosphere) L= Length of the cell, 38 cm

C= ozone concentration in parts per million (ppm)

I= UV light intensity of sample with ozone (sample gas)

I₀ = UV light intensity of sample without ozone (reference gas)

The sample is drawn into the analyzer through the sample bulk head and is split into two gas streams. One gas stream flows through an ozone scrubber to become the reference gas. The reference gas then flows to the reference solenoid valve.

The sample gas flows directly to the sample solenoid valve. The solenoid valves alternate the reference and sample gas streams between cells A and B in every 10 seconds. When cell A contains reference gas, cell B contains sample gas and vice versa.

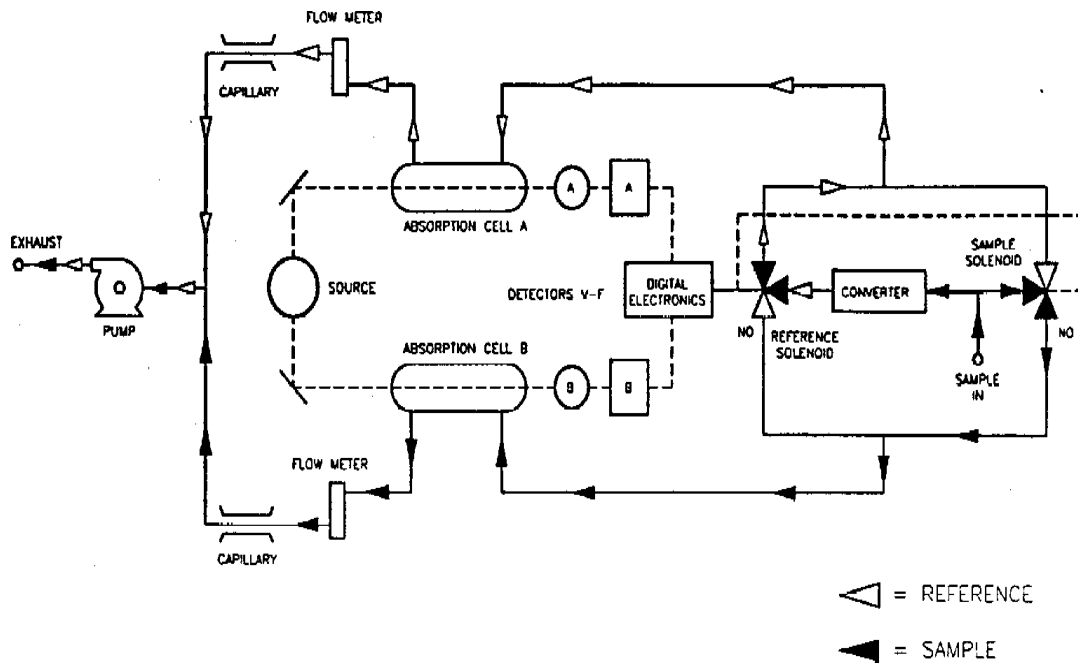


Figure 12.3: Schematic of UV Photometric Ozone Analyzer

The UV light intensities of each cell are measured by detectors A and B. When the solenoid valves switch the reference and sample gas streams to opposite cells, the light intensities are ignored for several seconds to allow the cells to be flushed. The analyzer calculates the ozone concentration for each cell and outputs the average concentration to the front panel display, the analogue outputs, and also makes the data available over the serial or Ethernet concentration. The analyzer is recalibrated periodically to check the accuracy of the measurements.

ii) SO₂ Analyzer:

Objective: Measurement of SO₂ gas concentration in air.

Principle:

SO₂ analyzer works on the principle of pulsed fluorescence analysis. SO₂ molecules absorb ultraviolet light and become excited, then decay to a lower energy state emitting UV light of another wavelength. The sample air is drawn into analyzer through sample bulkhead. The sample flows through a hydrocarbon kicker, which removes hydrocarbons from the sample by forcing the hydrocarbon molecules to permeate through the tube wall. Hydrocarbon kicker does not affect the SO₂ gas present in sample. The sample then flows into the fluorescence chamber, where pulsating UV light excites SO₂ molecules. As the excited SO₂ molecules decay to lower energy states they emit UV light that is proportional to the SO₂ concentration. The bandpass filter allows only the wavelength emitted by the excited SO₂ molecules to reach the photomultiplier tube (PMT); hence the PMT detects the UV light emissions from decaying SO₂ molecules. After leaving the optical chamber, the sample passes through the flow sensor, a capillary and the shell side of the hydrocarbon kicker. The sample then flows to the pump and exhausted out of the exhaust bulkhead of the analyzer.

Specifications:

(Model 43i designated by USEPA as a Reference method for SO₂ measurement)

Lower detectable limit	2 ppb (10 seconds averaging time) 1 ppb (60 seconds averaging time) 0.5 ppb (300 seconds averaging time)
Response time	80 seconds (10 seconds averaging time) 110 seconds (60 seconds averaging time) 320 seconds (300 seconds averaging time)
linearity	+/- 1 % of full scale
Sample flow rate	0.5 LPM (standard) 1.0 LPM (optional)
Operating temperature	15-30 °C

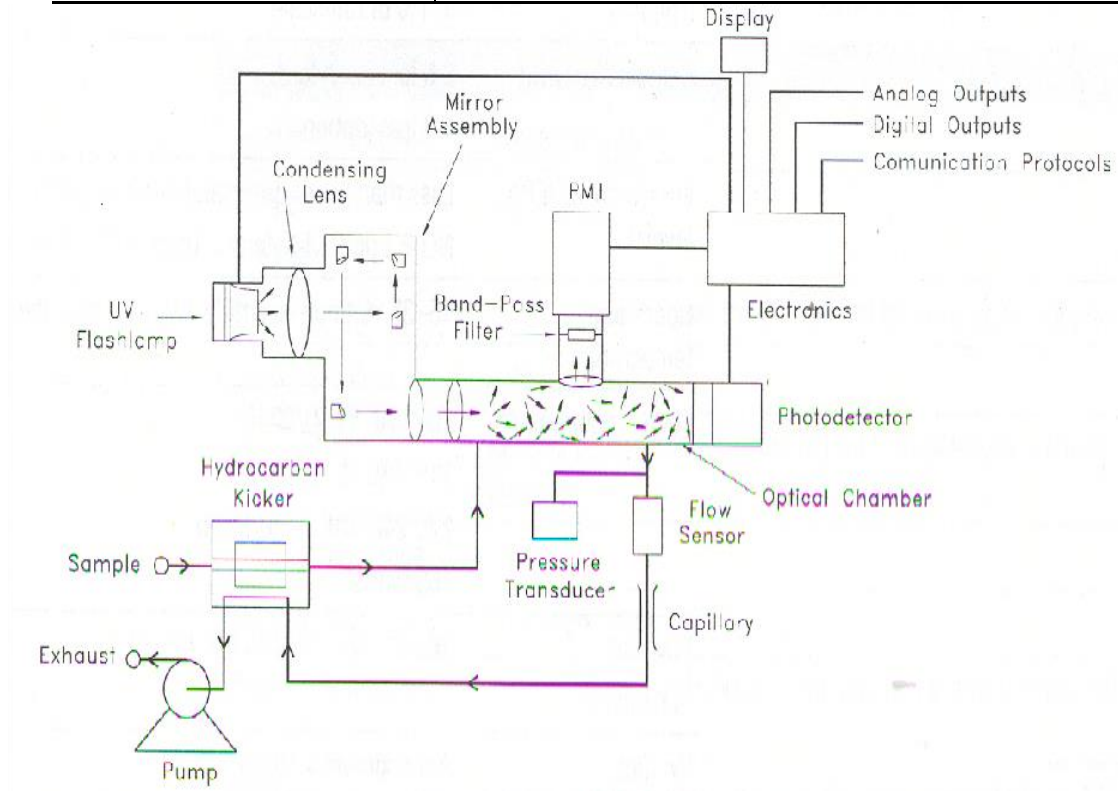


Figure 12.4: SO₂ Analyzer Model 43i flow schematics

Precautions:

1. Mirrors in the optical bench need not to be cleaned.
2. The instrument should be inspected occasionally for visible defects such as clogged Teflon lines and loose fittings.
3. Sample flow rate should not be less than 0.35 LPM.

iii) CO Analyzer:

Objective: CO analyzer measures CO concentration in air.

Principle:

CO analyzer measures CO concentration using Gas Filter Correlation (GFC). Working is based on the principle that Carbon monoxide (CO) absorbs infrared radiation at a wavelength of 4.6 micron.

The sample is drawn into the analyzer through the SAMPLE bulkhead and then flows through the optical bench. Radiation from an infrared source passed through a gas filter alternating between CO and N₂. The radiation then passes through a narrow band pass interference and enters the optical bench where absorption by the sample gas occurs. The infrared radiation then exits the optical bench and falls on an infrared detector.

Because infrared absorption is a nonlinear measurement technique, it is necessary for the instrument electronics to transform the basic analyzer signal into a linear output. Instrument uses an exact calibration curve to accurately linearize the instrument output over any range up to a concentration of 10,000 ppm.

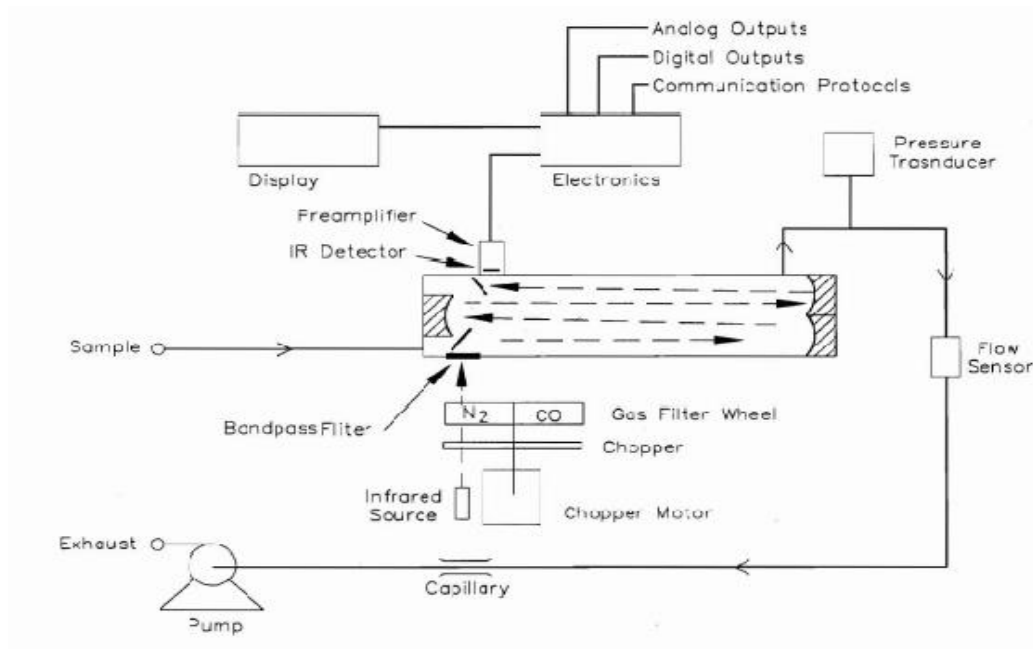


Figure 12.5: Schematic of Carbon-monoxide (CO) Analyzer

Specifications:

(Model 48i designated by USEPA as a Reference method for CO measurement)

Preset ranges	0-1, 2, 5, 10, 20, 50, 100, 200, 500, 1000, 2000, 5000, 10000 (ppm or mg/m ³)
Custom ranges	0-1 to 10,000 (ppm or mg/m ³)
Zero roise	0.02 ppm RMS (30 seconds averaging time)
Lower detection limits	0.04 ppm
Zero drift	<0.1 ppm
Sample flow rate	1.0 LPM
Response time	60 seconds (30 seconds averaging time)
Operating temperature	20-30 °C

Precautions:

1. Optics should be cleaned by using a cotton swab and methanol. Rinsing should be done by distilled or deionized water.
2. Fan Filter should be inspected and cleaned on regular basis.
3. IR source should be replaced, if IR intensity remains below 100,000 Hz

iv) Chemiluminescent NO-NO₂-NO_x Gas Analyzer:

Using chemiluminescence technology, the analyzer measures the amount of nitrogen oxides in the air from sub-ppb levels up to 100ppm. It is a single chamber, single photomultiplier tube design that cycles between the NO and NO_x modes. The available model 42i has independent outputs for NO, NO₂ & NO_x and each can be calibrated separately.

The available model 42i Chemiluminescence NO-NO₂-NO_x Gas Analyzer has the following features:

- High sensitivity
- Fast response time
- Independent NO-NO₂-NO_x range
- Linearity through all range.
- Replaceable NO₂ converter cartridge.

Principle:

The analyzer operates on the principle that nitric oxide (NO) and ozone (O₃) react to produce a characteristic luminescence with intensity linearly proportional to the NO concentration. Infrared light emission results when electronically excited NO₂ molecules decay to lower energy states.

Specifically:

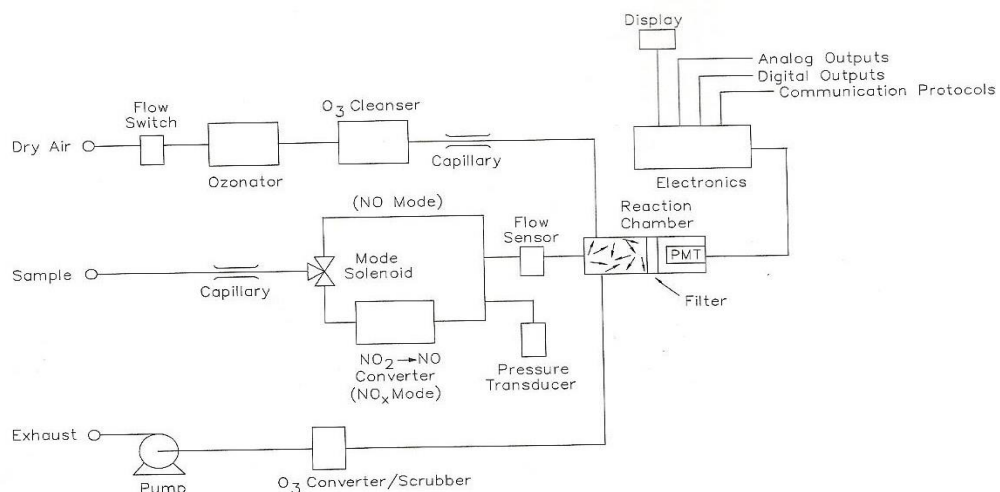
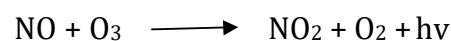


Figure 12.6: Schematic of Chemiluminescent Analyzer

NO₂ must first be transformed into NO before it can be measured using the chemiluminescent reaction. NO₂ is converted to NO by molybdenum NO₂ - to - NO

converter heated to about 325°C (the optional stainless steel convertor is heated to 625°C).

The ambient air sample is drawn into the analyzer through the sample bulkhead, the sample flows through a capillary, and then to the model solenoid valve. The solenoid valve routes the sample either straight to the reaction chamber (NO mode) or through the NO₂- to - NO converter and then to the reaction chamber (NO_x mode). A flow sensor to the reaction chamber measures the sample flow.

Dry air enters the analyzer through the dry air bulkhead, passes through a flow switch, and then through a silent discharge ozonator. The ozonator generates the ozone needed for the chemiluminescent reaction. At the reaction chamber, the ozone reacts with the NO in the sample to produce excited NO₂ molecules. A photomultiplier tube (PMT) housed in a thermoelectric cooler detects the luminescence generated during this reaction. From the reaction chamber, the exhaust travels through the ozone (O₃) converter to the pump, and is released through the vent. The difference between the concentrations is used to calculate the NO₂ concentration.